

I. EXTENSION OF THE TECHNIQUE OF
MICRORADIOGRAPHY TO BIOLOGICAL
MATERIALS

II. CRITICAL STUDIES OF SOME CONTROVERSIAL
THEORIES CONCERNING THE STRUCTURE OF CELLULOSE

BY
EDWARD JOHN BICEK
B.A., Carleton College, 1937

AN ABSTRACT OF A THESIS
SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS
FOR THE DEGREE OF DOCTOR OF PHILOSOPHY IN
CHEMISTRY IN THE GRADUATE SCHOOL OF
THE UNIVERSITY OF ILLINOIS, 1941

URBANA, ILLINOIS
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ACKNOWLEDGMENT

The author wishes to express his sincere appreciation to Professor George L. Clark under whose direction this work was carried out.

He also wishes to thank Doctors S. T. Gross, J. N. Mrgudich and W. F. Bradley, as well as all those with whom he worked, for stimulating discussions, assistance and advice during the course of these investigations.

PART I

EXTENSION OF THE TECHNIQUE OF MICRORADIOGRAPHY TO BIOLOGICAL MATERIALS

INTRODUCTION

Microradiography is the term applied to a technique in which the radiograph of a small object is registered on film of extremely fine grain size to give an image in which the radiographic details can be studied with the aid of a microscope. The practical applications of the method to metals and alloys have been pointed out by Clark and Shafer.¹ Applications to biological problems, on the other hand, are virtually untouched. Most significant has been the method of "histo-radiography" developed by Lamarque² and others which requires extremely thin sections, low voltages, vacuum cameras, and tubes of special design.

This study has been undertaken to develop a suitable microradiographic technique, using readily available equipment, which would be applicable to problems which lie between the domains of the sectioning and staining microscopic techniques and the usual radiographic methods, and to point out its possibilities and limitations.

EXPERIMENTAL

The contrast of radiographic images has been discussed theoretically with reference to the mass absorption coefficients and densities of the materials being radiographed, the response of the film to X-rays, the sensitivity of the eye to differences in density in the film, and the effect of scattered radiation. The resolution obtainable has been discussed with respect to the nature and thickness of the sample, the resolving power of the emulsion, the size of the source, and the relative positions of source, sample and film.

Ordinary diffraction tubes having low atomic number tar-

gets (Cr to Cu) and very transparent windows, and which operate at 20-30 kvp. have been shown to be a satisfactory and readily available source of soft (5-8 kv.) X-rays. Successful microradiographs of white oak sections, insect antennae, the eye and mouth parts of a fly, various other chitinous structures, and the section of the kidney of a frog which had been injected intravenously with colloidal ThO_2 are presented, which were obtained with this source of radiation and without the use of a vacuum camera.

Various experimental details such as alternative sources of radiation, protective coverings for the emulsion, filters to remove soft scattered radiation, film holders, developers and development times, methods for increasing the speed of the film, and the possibilities of stereoscopic microradiography are discussed. Gevaert and Eastman Lipmann emulsions were studied from the standpoint of speed, convenience in handling, grain size, and freedom from artifacts.

SUMMARY

The variables involved in the microradiography of biological materials have been studied and results are shown which indicate the applicability of a relatively simple technique to a wide variety of biological problems such as the study of thick, brittle, or opaque specimens; objects in which sectioning is impractical or in which the relation of the parts to the whole is desired; or in the study of physiological processes, with or without the aid of contrast media. Thick specimens with small differences in radiographic density, or samples containing large amounts of water, are not satisfactorily radiographed without more elaborate techniques.

PART II

CRITICAL STUDIES OF SOME CONTROVERSIAL THEORIES CONCERNING THE STRUCTURE OF CELLULOSE

INTRODUCTION

In the consideration of the X-ray diffraction effects produced by samples of native cellulose, explanations of the significance of the amorphous scattering, the small angle diffraction, and the broadening of the interferences have not been given as much attention and are not as clearly established as the explanation of the positions and relative intensities of the regular interferences which is given in terms of the crystal structure. An attempt has been made in this investigation to determine the significance of these non-crystalline diffraction effects and to evaluate the various theories concerning the particle size and role of amorphous matter in the structure of cellulose which have been put forward to explain the central scattering and broadening of the interferences.

RESULTS AND CONCLUSIONS

X-ray diffraction patterns of a large number of natural and synthetic polymers and of minerals known to possess defect structures have been prepared in order to study the factors governing the nature and occurrence of the central scattering. It has been pointed out by analogy to such materials as chrysotile and vermiculite and to copper-aluminum alloy systems that the central scattering may be due to randomness in the structure as well as to small particle size. Disagreements between the particle size of materials as determined by the theories of Guinier³ and Hosemann⁴ and as determined from the half-maximum breadth of the interferences are pointed out as indicating that the two methods probably do not measure the same phenomenon.

Vacuum-monochromatic patterns have been taken of white oak, ramie, flax and jute. The patterns show a relatively

small amount of amorphous scattering, and microphotometer traces along the equator indicate that samples which give the broadest interferences would have the largest particle size on the basis of Guinier's calculation from the central scattering. This was taken to indicate that the central scattering and the broadening of the interferences cannot both be due to particle size effects.

It has been suggested that cellulose and polymers in general may have a statistical randomness in the orientation of the repeating units throughout the crystalline regions, that the alternative positions are nearly but not exactly equivalent, and that periodicities in the structure arising from these statistical deviations from the ideal arrangement are the cause of at least part of the small angle scattering. It is noted that the accepted structure of cellulose can be considered as a statement of the average arrangement to be expected on such a basis.

It has also been pointed out that the periodicity due to such a randomness should not be considered synonymous with effects due to actual physical discontinuities, since materials which possess such lattice imperfections can then have both structural "particle size" and physical discontinuity "particle size." The problem is similar to the indistinguishable effects of particle size and strain on the broadening of the interferences. It is possible that a consideration of both the broadening of the interferences and the small angle scattering may eventually lead to an independent evaluation of lattice imperfections, strain, and particle size, as well as to a clearer concept of the structure of cellulose and other polymers.

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VITA

The author was born August 17, 1915 in Tracy, Minnesota and received his primary and secondary education in the public schools there. After graduation in 1933 he entered Carleton College and in 1937 received the Bachelor of Arts degree, Cum Laude, and with honors in chemistry. In the fall of the same year he entered the Department of Chemistry of the Graduate School of the University of Illinois. From 1937 to 1940 he held a teaching assistantship in the Analytical Division and was granted a University Fellowship for the year 1940-1941.

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